

**Microwave Energy Effects on Polymer-Matrix and Fibre Reinforcement of Composites during Bonding Process by Microwaves**

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**Abstract:** One important thing to be studied in the bonding of thermoplastic composite material using microwave irradiation is the effects of microwave energy on the matrix and fibre reinforcement of the composite. In this research, thirty three percent by weight random glass fibre reinforced polystyrene [PS/GF (33%)] is chosen for the study. Microscopy study is used to find out the microstructural characteristics along and around the interface of the welds. The separation distance of the reinforcing carbon filaments was varied and heat transfer in the material during joining was studied. The reasons why the thermoplastic matrix composite materials were weakened by prolonged microwave irradiation were also studied and analysed.

## **Introduction**

The use of microwave energy in joining thermoplastic matrix composite materials has been well documented [1-9]. This research starts with the concept of joining fibre reinforced thermoplastic (FRTP) matrix composite material using microwave irradiation with primers. The composite material used is PS/GF (33%) which was specially manufactured in Brisbane, Australia. The matrix of the composite, polystyrene, PS, is poly (phenylethene) with repeating units of  $C_2H_3.C_6H_5$  [10]. The reinforcement was made 33% by weight because thirty three percent by weight random glass fibre reinforced Nylon 66 [Nylon/GF (33%)] is readily available in market and can later be used for comparison. The joints, with araldite as primer, were then lap shear tested and it was found that joints with Araldite as a primer cured by microwave irradiation were stronger than those obtained by curing the primer at ambient conditions. This led to the idea that the Araldite might have diffused into the thermoplastic matrix. In order

to prove this, two surface analysis procedures were carried out. One was microscopy inspection and the other was x-ray photoelectron spectroscopy. Both analyses confirmed that diffusion had taken place [9]. This paper discusses the effects of microwave energy on the matrix and fibre reinforcement of thermoplastic composite during bonding process. The first is the effects of microwave energy on the structure of the matrix, the polymer. The other is the relationship between the reinforcing filaments, eg, glass, carbon or aramid fibre filaments, and microwave energy. The material will be used in the manufacture of consumer products, where polymer without reinforcement is not strong enough to fulfil their functions. Microwave processing of this material provides an alternative for enhancing material properties as well as economic advantages through energy savings and accelerated product development.

### **Polymers and Their Structures**

The basic unit of polymer is the molecule or, more accurately, the macromolecule, which is composed of thousand of atoms. The spine of these molecules is the carbon chain of atoms and there are three basic types of polymeric materials. These are linear, space network and elastomeric polymers and are shown in Figure 1 [11, 12]. The *linear structure* is a chain that is not a straight line but more like spaghetti [11]. These molecules slide by each other upon heating and form the thermoplastic polymers. The *space network structure* is rigid and makes up the thermosetting polymers. The coiled structures exhibit elastic extension of as much as 1000% and are aptly termed elastomers. The molecular chains of a polymer may be completely tangled up or can be arranged in an orderly manner. If the chains are tangled up, the polymeric materials are

said to be amorphous, otherwise they are crystallised [11]. The thermoplastic matrix used in this research is polystyrene, PS, which has a degree of crystallinity of 0% and therefore couples better with microwave energy than lower density polyethylene, LDPE, which has a crystallinity of 60 – 95 % [13, 14]. This is further confirmed by the better dielectric properties, especially loss tangent, of PS over those of PE. The loss tangent of PS ( $5.3 \times 10^{-4}$ ) at microwave frequencies is 1.5 times higher than that of LDPE ( $3.6 \times 10^{-4}$ ) at the same frequencies as listed in Table 1 [15].

### **Microwave Irradiation and the Matrix**

In this research, polystyrene (PS) is used as the matrix of the FRTP composite because it is generally flexible and relatively soft and if heated they become softer and more flexible. Their behaviour is totally due to their linear structure, in which the chain of each linear polymer slides over each other. In the joining of PS/GF (33%) using microwave energy with Araldite as primer, it is expected that the Araldite will absorb more microwave energy than the PS/GF (33%) and convert it into heat [9]. The composite material will not absorb microwave energy without the use of the primer. The heat will initially heat up the primer and the heated primer will then conduct the heat into the composite. The matrix of the composite is a thermoplastic resin, PS with linear structure. The percentage of free volume is therefore high and the chains of each linear polymer slide over each other easily with the limited supply of conducted heat. This is proved by the microscopy study [9, 11].

PS/GF (33%) was joined using microwave energy with Araldite as primer at a power level of 800W, a frequency of 2.45 GHz and exposure times of 45 and 60 seconds. First, the sample with an exposure of 45 seconds to microwave irradiation was studied under Scanning Electron Microscope (SEM). At 100 times magnification, it was found that the bondline was observed to have black colour, while the parent material in the matrix was grey. This is shown in Figure 2. There were spots of glass fibre filaments that appeared as bright spots on the interface (bondline). Such spots were found to be numerous, as expected, in the parent material matrix. At 400 times magnification, it was found that the bondline occupied most of the view of the microscope. The glass fibre filaments appeared as bright patches in the bondline. The structure of the parent material adjacent to the bondline was the same as that which was observed away from the bondline. This is shown in Figure 3 (as indicated by arrows). No change of phase was inferred. However, since glass fibre filaments were present in the bondline, it could be argued that they had been flushed due to flowing of the melting parent material, ie PS/GF (33%) into the Araldite, primer. Therefore, a change in phase, from solid to liquid, of the parent materials just adjacent to the interface (bondline) was observed (Figure 3). X-ray Photoelectron Spectroscopy also showed the diffusion of the parent material into the Araldite and vice-versa, which in turn confirmed the argument that there was flow of parent material towards the bondline and also a change of phase in the parent material [9].

Secondly, the sample with an exposure time of 60 seconds was studied. At 100 times magnification, similar microscopic characteristics were observed and are shown Figure 4. At 400 times magnification, in addition to the microscopic view

as in the 45-second exposure time, it was found that the colour and structure of the parent material just adjacent to the bondline were different to that observed away from the bondline. This implied that a phase change of parent material had taken place. This is shown in Figure 5 and the change in phase and the rise in temperature of the parent material has changed the loss tangent of the material. It was experimentally found that the higher the temperature the higher the value of the dielectric loss [16-18]. As a result of larger amounts of energy absorbed and converted to heat, the whole test piece became warm and pliable at the end of the joining process. In conclusion, microwave irradiation of the primer and the parent material brought about hybrid heating by the combination of microwave energy induced and thermal conduction that took place along the interface. This hybrid heating subsequently caused a phase change in the matrix, which became more susceptible to microwaves and thus hot spots occurred at macrolevel (Figure 5).

Thirty three percent random glass fibre reinforced polystyrene was then joined using variable frequency microwave (VFM) energy with Araldite as primer at a power level of 200W, a central frequency of 7.25 GHz and a bandwidth of 1.1 GHz and exposure times of 420 and 450 seconds [10]. First, the sample with an exposure time of 420 seconds to VFM irradiation was studied. At 100 times magnification, it was found that the bondline was observed to have black colour, while the parent material in the matrix was grey. This is shown in Figure 6. Only very few glass fibre filaments appeared on the bondline. At 400 times magnification, some glass fibre filaments were found as bright patches on the bondline as depicted in Figure 7. At 1000 times magnification, the glass fibres

are more obvious and this is shown in Figure 8. The microscopic observations found were similar to those found in Figures 2 and 3 respectively except that less glass fibre filaments on the bondline inferred that diffusion of the parent material to the araldite and vice-versa was not so obvious as in the fixed frequency case. Four hundred and twenty seconds are the maximum exposure time of the composite to VFM before the parent material is damaged [10]. Next, the sample with an exposure time of 450 seconds to VFM energy was investigated and it was at this time of exposure that the parent material was weakened [10]. At, 100, 400 and 1000 times of magnifications, similar microscopic characteristics were observed as in the case of an exposure time of 420 seconds and were shown in Figures 9, 10 and 11 respectively. In both cases, no change of phase was inferred. However, since more glass fibre filaments were present in the bondline, it could be argued that they had been flushed due to flowing of the melting parent material, ie PS/GF (33%) into the Araldite, primer. Therefore, a change in phase, from solid to liquid, of the parent materials just adjacent to the bondline was observed. The parent material, PS/GF (33%), however, did not present a second phase as in the case of Figure 5. This implied that the power level used 200 W, was not high enough to cause such a change to take place. To improve the situation, a machine with higher input power, eg Microcure 2100 model 2000 of 2000 W will be required.

### **Microwave Irradiation and the Reinforcing Filament**

The next theme to be discussed is the relationship between microwave energy and the reinforcing filaments, eg, glass, carbon or aramid fibre filaments. The

loss tangent of glass fibre filaments is very low, 0.0002, and it does not increase much with rising temperature [15, 17-20]. They will not be discussed in detail. On the other hand, the dielectric loss of carbon fibre filaments is expected to be much higher than that of glass and aramid fibre filaments and its importance in the joining process is therefore investigated in details. Two carbon fibre filaments of 6-mm length used in the manufacture of the FRTP composites were laid down on a test piece [of PS/GF (33%)] used in the research and positioned side by side as shown in Figure 12. They were held in positions by smearing extremely small amount of glue of low loss tangent to one end of the filament. The separation distance of the two filaments,  $d$  mm, was varied from 1mm to 4 mm in steps of 1 mm. They were then placed in the waveguide front of the fixed frequency (2.45 GHz) microwave facility.

It was found that, irrespective of the distance of separation of the filaments,  $d$  mm, the carbon filaments started to arc after exposure to 800 W power level microwave irradiation for 12 seconds. If the exposure time was increased to 15 seconds or more, conduction of heat from the carbon filaments to their surrounding area took place quickly and the surface of the test piece was burnt and became black. Heat was also conducted to the other end of the test piece, which becomes warm. If the exposure time was made 13 or 14 seconds, the test piece was slightly blackened around the tips of the carbon fibre filaments and the radius of the blackened part was 0.5 to 1 mm. This is shown in Figure 13.

Referring to Figure 13 and considering the black patch. It can be deduced that the carbon filament heated the matrix of the composite, PS, to its melting point,



240 °C and continued to supply heat until it became charred [21]. This means that the bonds in the PS were broken leaving carbon behind. The black patch proved by X-ray Photoelectron Spectroscopy to be carbon formed from the breaking of the C-C, C-H and C=C bonds of PS.

In the surface analysis using X-ray Photoelectron Spectroscopy, two samples of PS/GF (33%) were used. One is a sample of PS/GF (33%) with black patches on it and the other is a clean PS/GF (33%) sample used as control. Referring to Figure 14, it is found that the elements present in the sample of PS/GF (33%) with black patches are oxygen, carbon and silicon. This is similar to the elements found in the control sample as illustrated in Figure 15. The intensity of carbon in Figures 14 and 15 are fitted into the curves of Figures 16 and 17 respectively. Referring to Figure 17, it is found that there is only one peak for the intensity of carbon atoms, which represents carbon atoms in C-H and C-C bonds in PS/GF (33%). Referring to Figure 16, it is found that there are two peaks for the intensity of carbon atoms; one represents carbon atoms in C-H, C=C and C-C bonds in PS/GF (33%) and the other independent carbon atoms (graphite). This concluded that independent carbon atoms exist in the black patches.

Referring to Figure 13, the diameter of the carbon fibre is 1.0 mm and its length is 6 mm. Its volume =  $\pi/4 \times (1.0)^2 \times 6 \text{ mm}^3 = 4.712 \text{ mm}^3$  or  $4.712 \times 10^{-3} \text{ cm}^3$ .

Since the density of the carbon fibre is  $1.86 \text{ g/cm}^3$ , the mass of the carbon fibre [22]

= volume x density

$$= 4.712 \times 10^{-3} \text{ cm}^3 \times 1.86 \text{ g/cm}^3 = 8.764 \times 10^{-3} \text{ g.}$$

The specific heat capacity of the carbon fibre is 0.61 J/gK and the energy absorbed by the carbon fibre by bringing it from room temperature (20 °C) to 240 °C [23]

= mass x specific heat x change in temperature

=  $8.764 \times 10^{-3} \times 0.61 \times (240-20) = 1.176 \text{ (J)}$ , which was the energy transferred to the thermoplastic matrix, assuming little loss to the surrounding.

Referring to Figure 13, the diameter of the patch is 1.0 mm and assume its depth is 0.005 mm. Its volume =  $\pi/4 \times (1)^2 \times 0.005 = 3.927 \times 10^{-3} \text{ mm}^3 = 3.927 \times 10^{-6} \text{ cm}^3$ .

Its mass = density x volume =  $1.05 \times 3.927 \times 10^{-6} = 4.123 \times 10^{-6} \text{ g}$ .

In a mer of PS, ie, phenylethene,  $\text{C}_2\text{H}_3.\text{C}_6\text{H}_5$  there are one C-C bond, three C-H bonds and one C-benzene ring bond, which consists of four C-C bond, three C=C bonds and five C-H bonds. The energy required to break one-gram mole of the C-C bond is 334.88 KJ, that of the C-H bond is 443.716 KJ and that of the C=C bond is 145 KJ [11].

The energy required to break the 5 C-C bond (black patch) in Figure 13

$$= 5 \times \frac{4.123 \times 10^{-6}}{104} \times 334.88 \text{ KJ} = 0.06638 \text{ J} .$$

The energy required to break the 8 C-H bond (black patch)

$$= 8 \times \frac{4.123 \times 10^{-6}}{104} \times 443.716 \text{ KJ} = 0.1407 \text{ J} .$$

Similarly, energy required to break the 3 C=C bond (black patch)

$$= 3 \times \frac{4.123 \times 10^{-6}}{104} \times 145 \text{ KJ} = 0.01724 \text{ J}$$

Total energy required to break all the bonds of the PS (charred area)  
= 0.06638 J + 0.1407 J + 0.01724 J = 0.2243 J.

The energy transferred from the carbon fibre to the matrix of the composite was found to be 1.176 J, which was more than enough to break the bonds (0.2243J) of the PS mer and char it.

In joining thirty three percent by weight random carbon fibre reinforced low density polyethylene [LDPE/CF (33%)] with a fixed frequency of 2.45 GHz and at the power level of 400W, it was found that the material started to arc after being exposed to microwave irradiation for 8 seconds [24]. Again, in joining PS/CF (33%) with a fixed frequency of 2.45 GHz and at the power level of 400 W, the composite started to arc after being exposed to microwave energy for 11 seconds [25]. In short, the composite materials started to arc in less than 11 seconds when exposed to the power of 4000 W. Comparing these results with those of the independent carbon fibre filaments' study in this research, it was found that the composite material, PS/CF (33%) coupled better with microwave irradiation. The reason behind this is that the filament-to-filament distance in the FRTP composites is much less than 1. The filaments, once heated, conducted heat in their respective neighbourhood easily and in addition, in the closely packed FRTP composites (33% by weight of fibre filaments), some of the filaments may have touched one another which may have led to d.c. conduction, both of which will heat the composites up and cause arcing at a much lower power level and time of exposure. On account of the above reasons and when the strength of the

composite can be compromised, it can be argued that the amount of carbon fibre filaments in the composites can be reduced to cut the cost of the materials, which at the same time can couple with microwave irradiation more efficiently.

The last topic to be discussed concerns about the weakened parent materials observed. The composite materials, PS/CF (33%) and LDPE/CF (33%) were weakened by excessive exposure to VFM irradiation. The discussion is split into two parts; one part deals with the effect of VFM energy on the reinforcement of the composite, ie the carbon filaments, while the other is concerned with that in the matrix of the composite, ie the thermoplastic.

Considering the joining of PS/CF (33%) using VFM with no primer the power used was 100 W, the maximum exposure time was 120 seconds and the maximum temperature reached was 95°C, which was higher than the heat-distortion temperature (82 °C) of PS and the temperature limit in air for carbon filaments impregnated in polystyrene (93°C) [11, 26]. The matrix and the reinforcement of the composite were damaged. The bonds of the matrix, PS, were broken, leaving behind black carbon as mentioned in the previous paragraphs. The lap shear strength values obtained for PS/CF (33%) (222 N/cm<sup>2</sup>) were lower than those procured for PS/GF (33%) (331 N/cm<sup>2</sup>) by around 109 N/cm<sup>2</sup> and this provided an extra proof that the carbon fibre filaments were damaged by prolonged VFM irradiation because the carbon fibre filament (3100 MP<sub>a</sub>) is stronger than the glass fibre filament (2200 MP<sub>a</sub>) [10, 22]. During the joining process, the carbon fibre filament coupled very well with microwave energy and was heated directly which cause damage to occur. On the other hand,

the glass fibre filament did not absorb microwave irradiation directly but it was heated by the Araldite through thermal conduction. In addition, the oxidation temperature (505°C) of the carbon fibre filament is also closer to the maximum processing temperature (95°C). As a result, the carbon fibre filament was damaged and became brittle. All of the above strength data were measured by the author some time ago, who was unable and to find similar data from other's work.

Considering the joining of thirty three percent by weight random carbon fibre reinforced low density polyethylene [LDPE/CF (33%)] using VFM at fixed frequency of 2.5 GHz with no primer, the power used was 100 W, the maximum exposure time was 20 seconds and the maximum temperature reached was 95°C, which was higher than the heat-distortion temperature (49 °C) of LDPE, and the temperature limit in air for carbon filaments impregnated in polystyrene (93°C) [11, 23]. The matrix and the reinforcement of the composite were damaged. The bonds of the matrix, PS, were broken, leaving behind black carbon as mentioned in the previous paragraphs. Since fixed frequency had been used in the processing, hot spots had also contributed to the damage of the matrix in spite of its short exposure to microwave irradiation. The peak lap shear strength value obtained for LDPE/CF (33%) were lower than those procured for LDPE/GF (33%) by 16 N/cm<sup>2</sup> and this provided an extra proof that the carbon fibre filaments were damaged by prolonged VFM irradiation because the carbon fibre filament (3100 MP<sub>a</sub>) is stronger than the glass fibre as mentioned previously [10]. Other arguments are similar to the case of PS/CF (33%).

## **Conclusion**

In the joining of thermoplastic matrix composite materials using microwave irradiation, it is found that some characteristics, eg low crystallinity favour the process while others hinder it. Glass fibre filaments are not damaged by microwave heating, while carbon fibre filaments were weakened by prolonged exposure to microwave irradiation. The resins of polymers used in this research were thermoplastics but it could be argued that the process could be similarly used in processing thermosetting resin matrix composite materials because as technology advances, materials have been developed which evade the above classification. Mixtures of thermoplastic and thermosetting resins have been developed in which the thermoplastic material acts as a toughening agent for thermosetting resin [27].

The merits of employing microwaves in joining thermoplastic composites include having a clean and reliable interface at the joints and fast joining time with minimum destruction of the properties of the bulk materials. Limitations encountered in other processes are avoided [28]. Microwave characteristics that are not available in conventional processing of materials include: penetrating radiation; controllable electric field distribution; rapid heating; selective heating of materials and self-limiting reactions. Alone or in combination, these characteristics can lead to benefits and opportunities.

The above characteristics also present unfavourable effects in materials processing. First, due to inadequate penetration of the microwave energy, bulk

materials with significant ionic or metallic conductivity cannot be efficiently processed. Second, on account of their limited absorption of the incident power, insulators with low dielectric loss are difficult to heat from room temperature to the required temperature. Lastly, materials with loss factors that vary significantly with temperature during processing will often lead to hot spots and thermal runaway [14].

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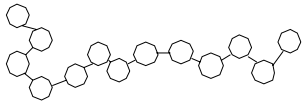
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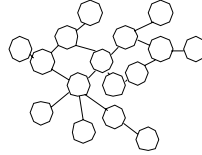
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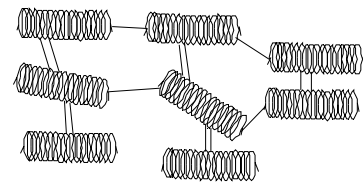
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LINEAR THERMOPLASTIC  
POLYMERS

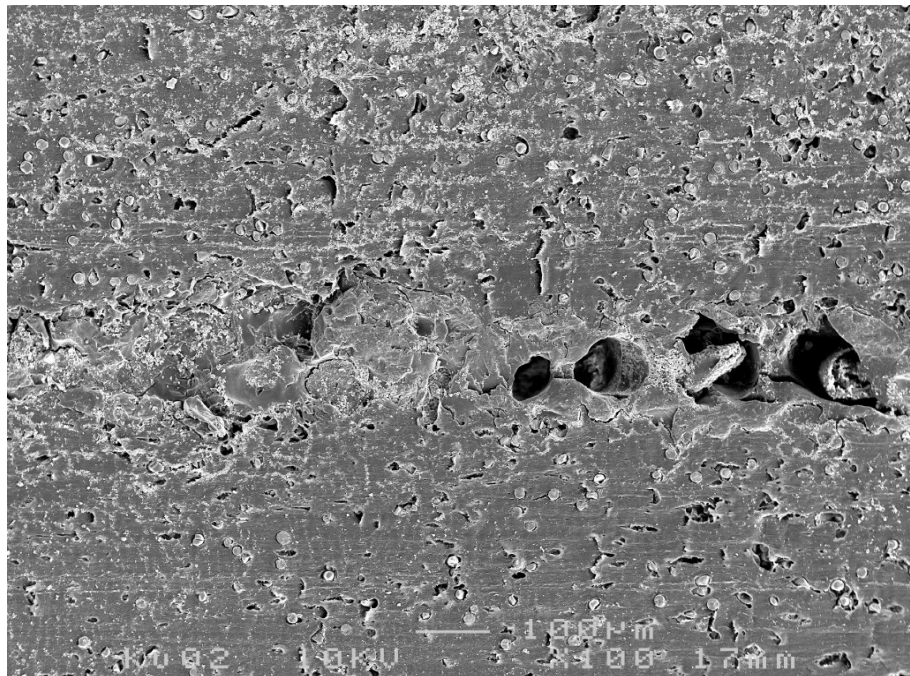


SPACE STRUCTURES -  
THERMOSETTING  
POLYMERS

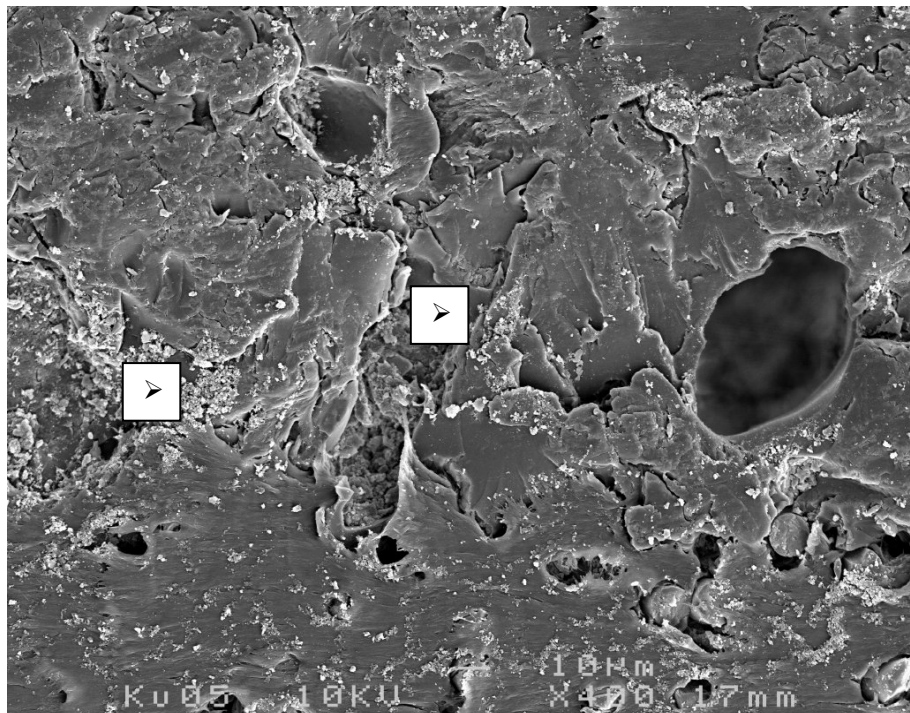


ELASTOMERS

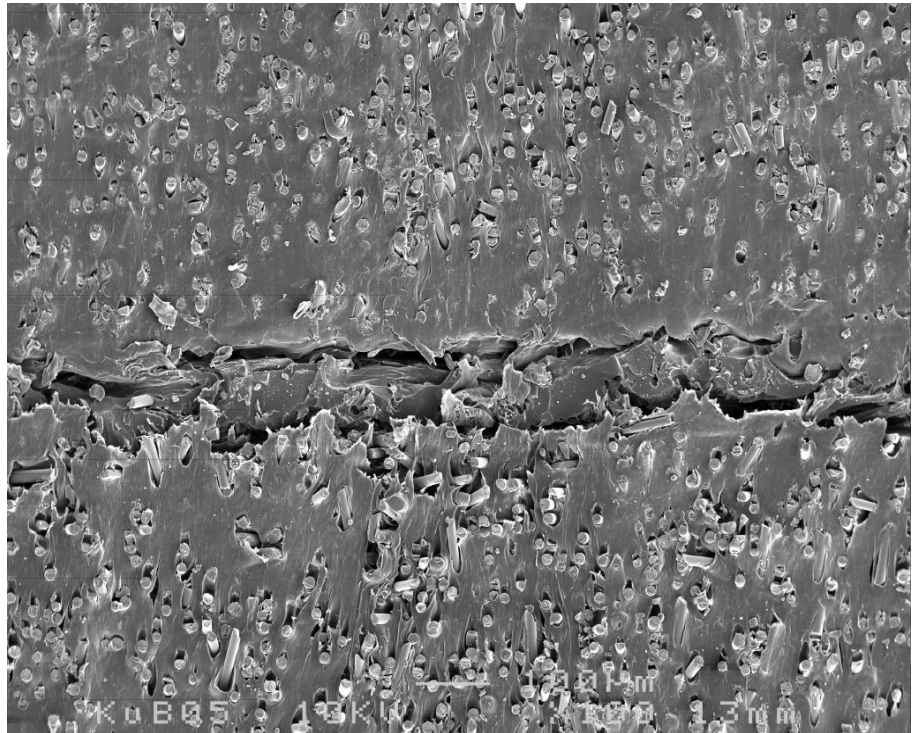
**Figure 1: Three Types of Basic Polymeric Materials**



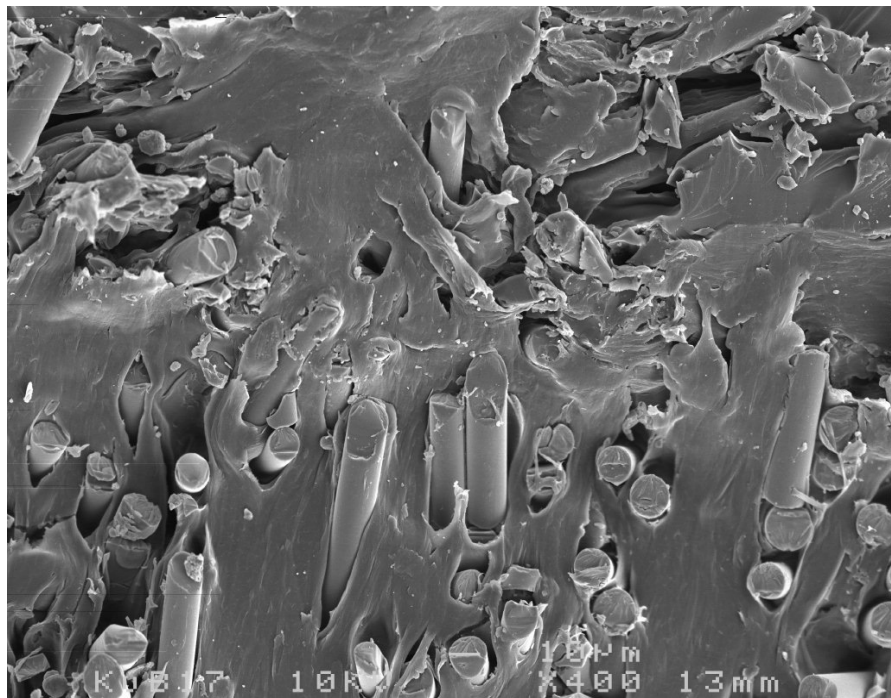
**Figure 2: Lap Joint Bondline of PS/GF (33%) at a Magnification of 100 Times with Joining Parameters of 2.45 GHz 800 W and 45 seconds**



**Figure 3: Lap Joint Bondline of PS/GF (33%) at a Magnification of 400 times with Joining Parameters of 2.45 GHz 800 W and 45 seconds**

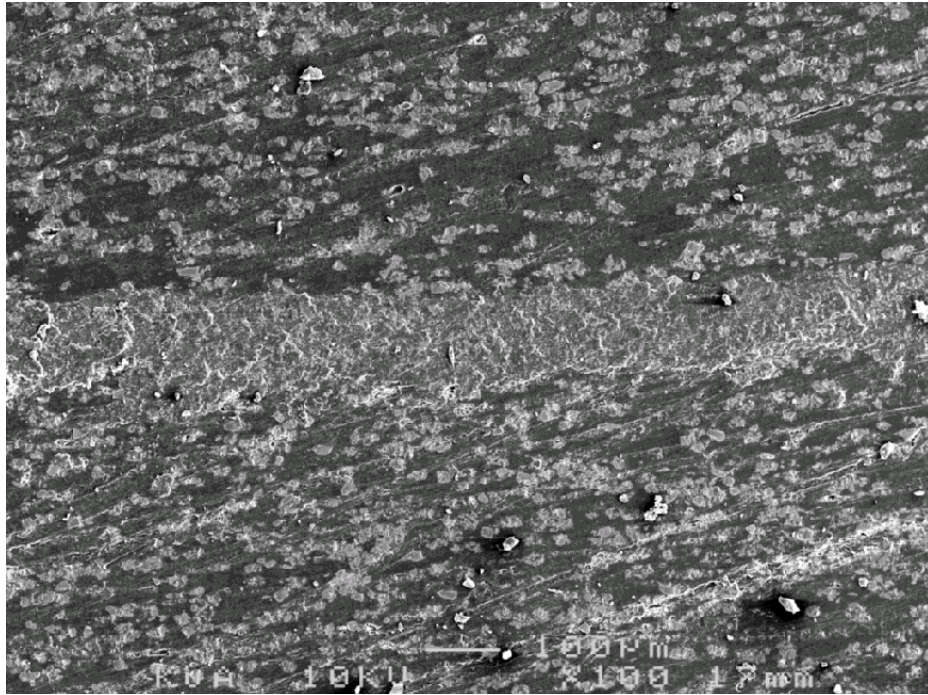


**Figure 4: Lap Joint Bondline of PS/GF (33%) at a Magnification of 100 Times with Joining Parameters of 2.45 GHz 800 W and 60 seconds**

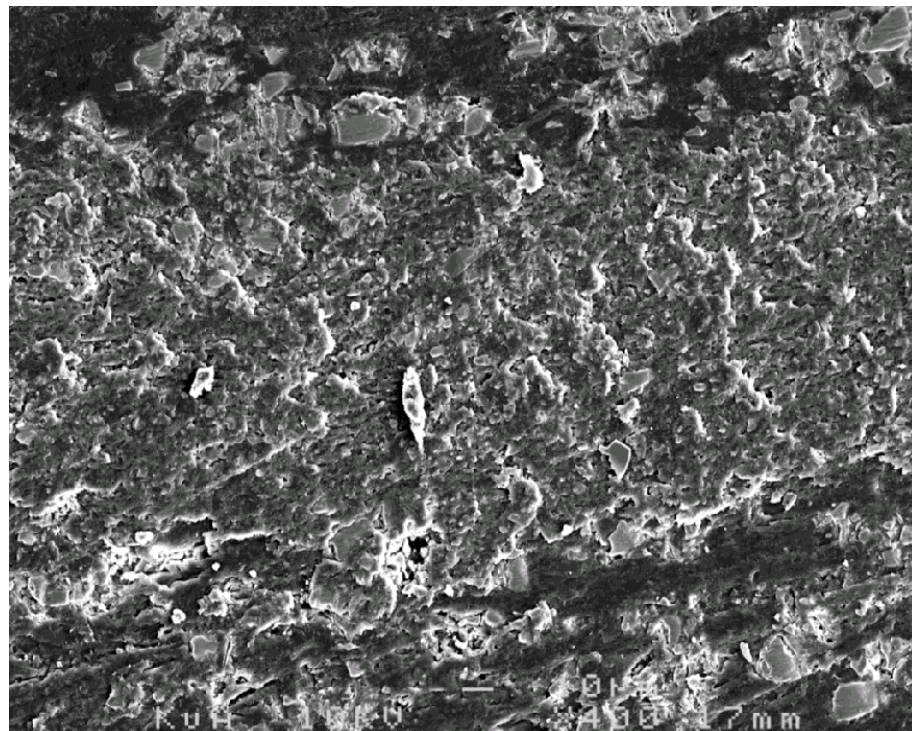


**Figure 5: Lap Joint Bondline of PS/GF (33%) at a Magnification of 400 Times with Joining Parameters of 2.45 GHz 800 W and 60 seconds**



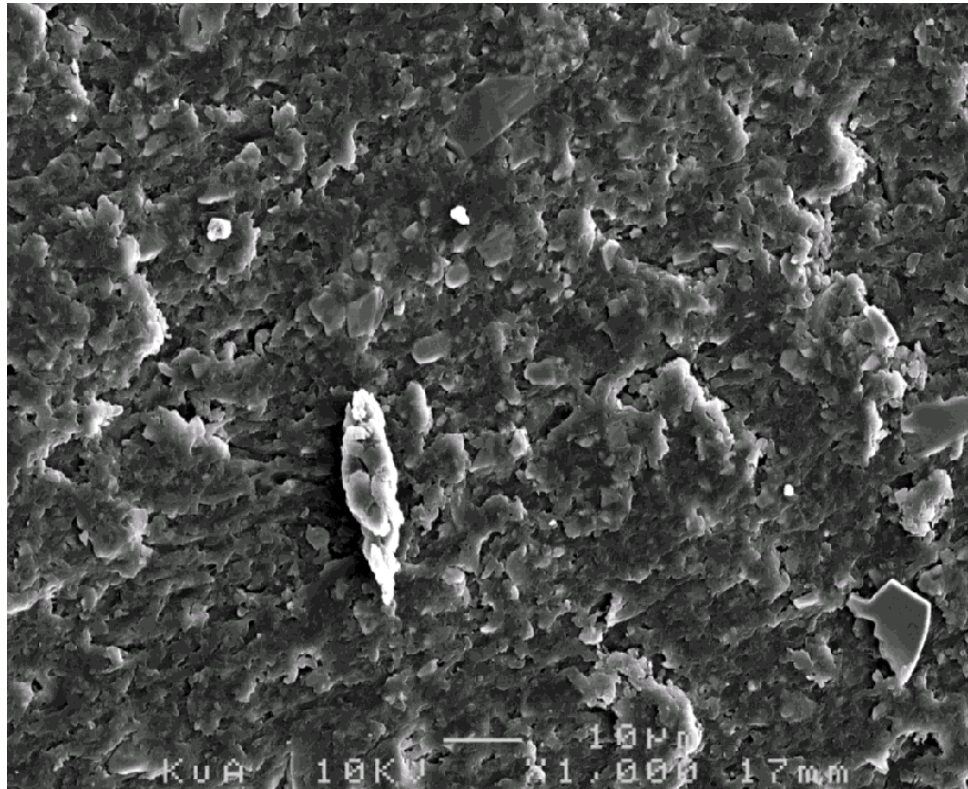


**Figure 6: Lap Joint Bondline of PS/GF (33%) at a Magnification of 100 Times with Joining Parameters of VFM, 200 W and 420 seconds**

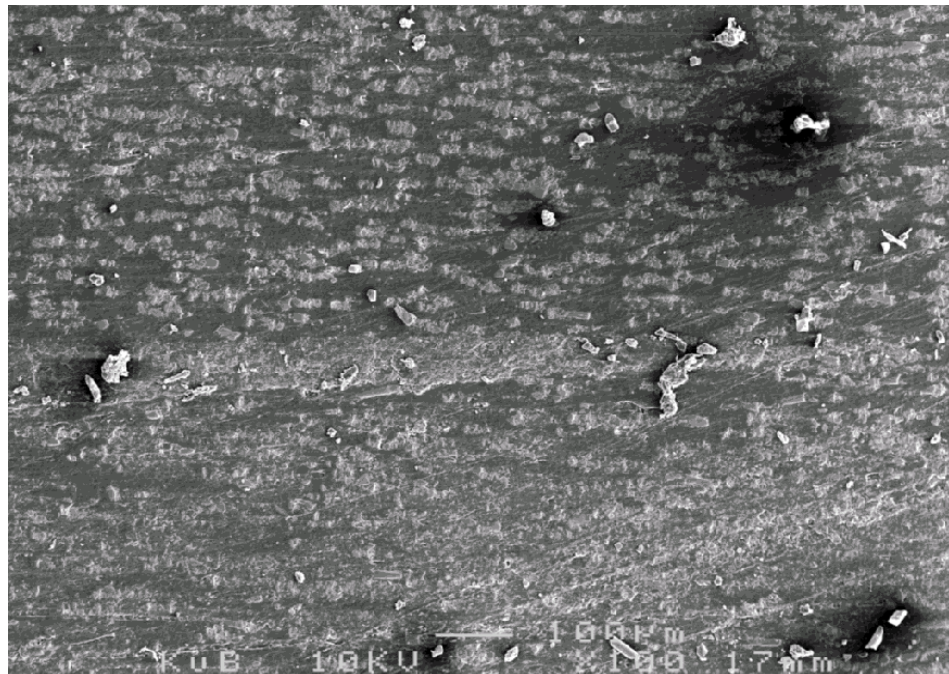


**Figure 7: Lap Joint Bondline of PS/GF (33%) at a Magnification of 400 Times with Joining Parameters of VFM, 200 W and 420 seconds**



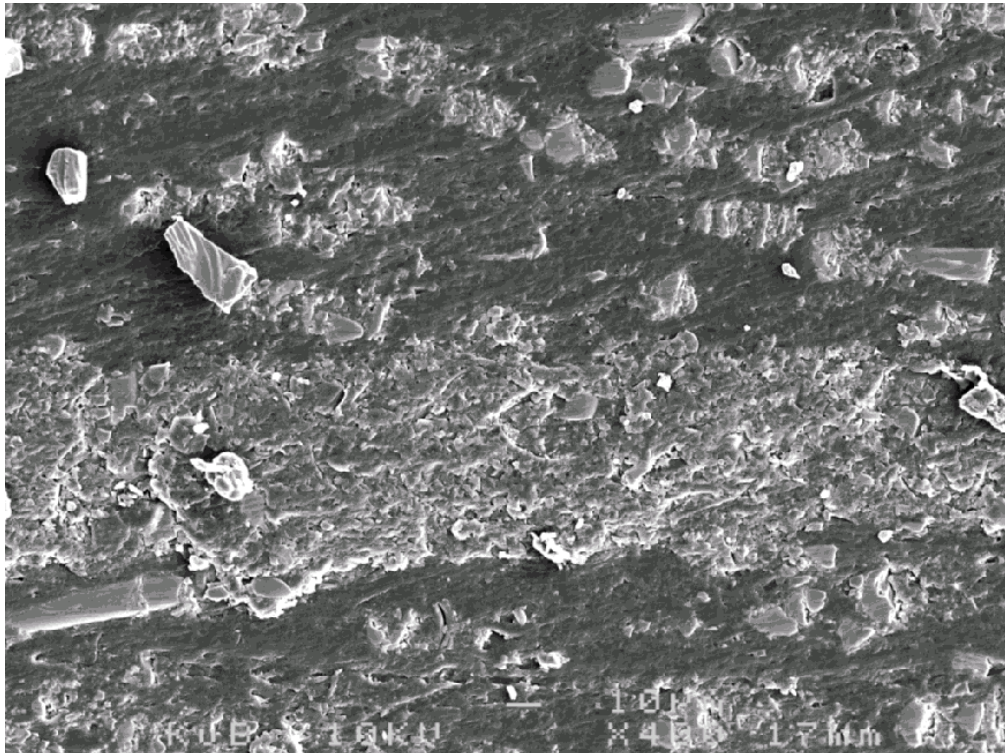


**Figure 8: Lap Joint Bondline of PS/GF (33%) at a Magnification of 1000 Times with Joining Parameters of VFM, 200 W and 420 seconds**

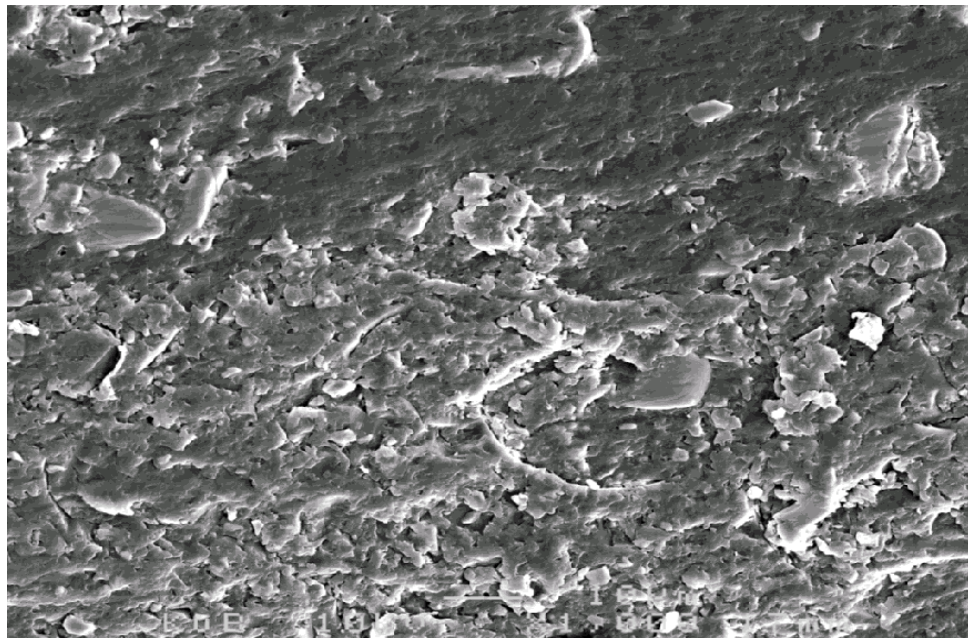


**Figure 9: Lap Joint Bondline of PS/GF (33%) at a Magnification of 100 Times with Joining Parameters of VFM, 200 W and 450 seconds**

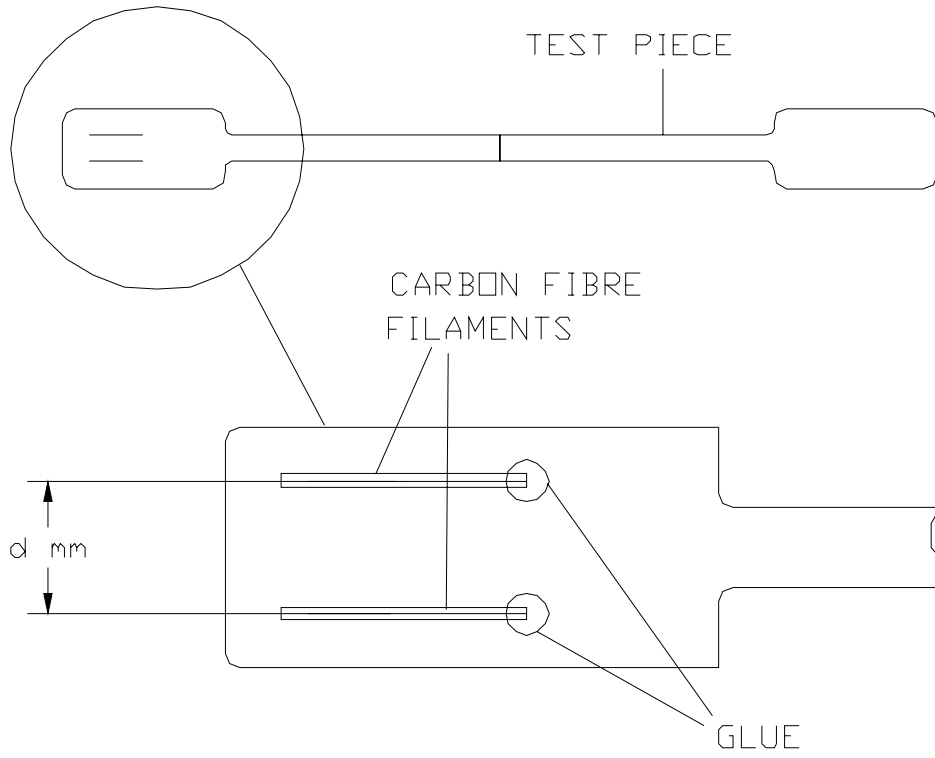




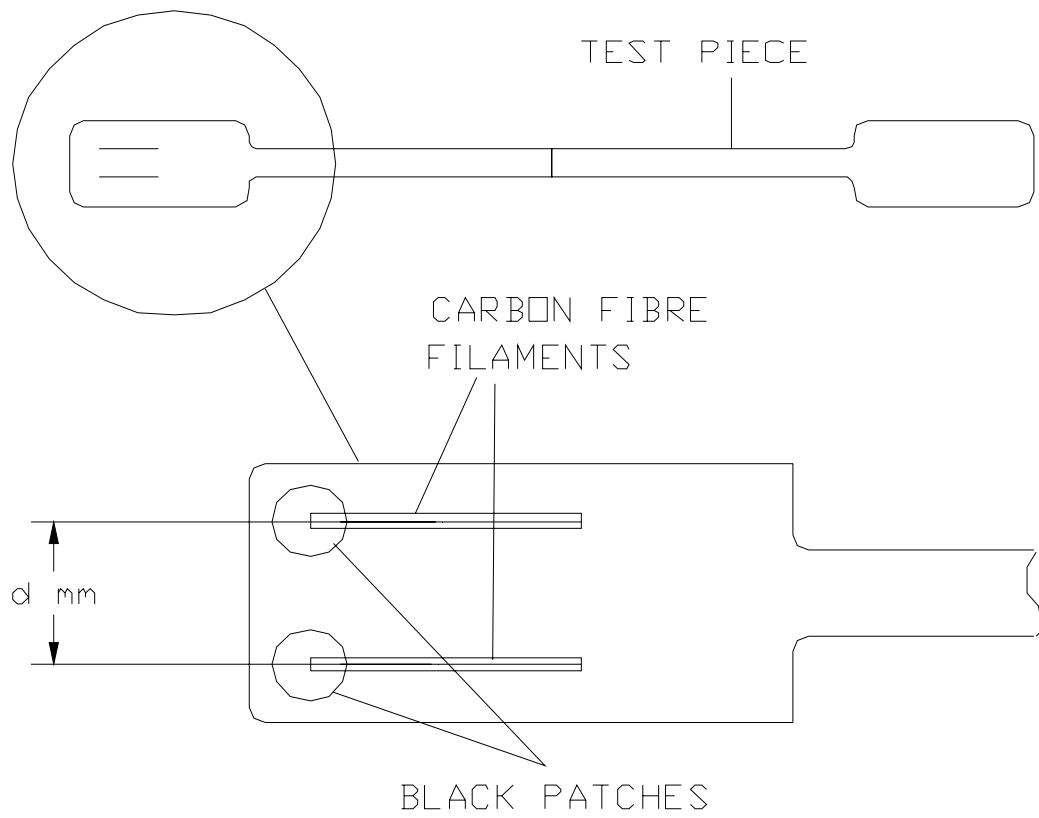
**Figure 10: Lap Joint Bondline of PS/GF (33%) at a Magnification of 400 Times with Joining Parameters of VFM, 200 W and 450 seconds**



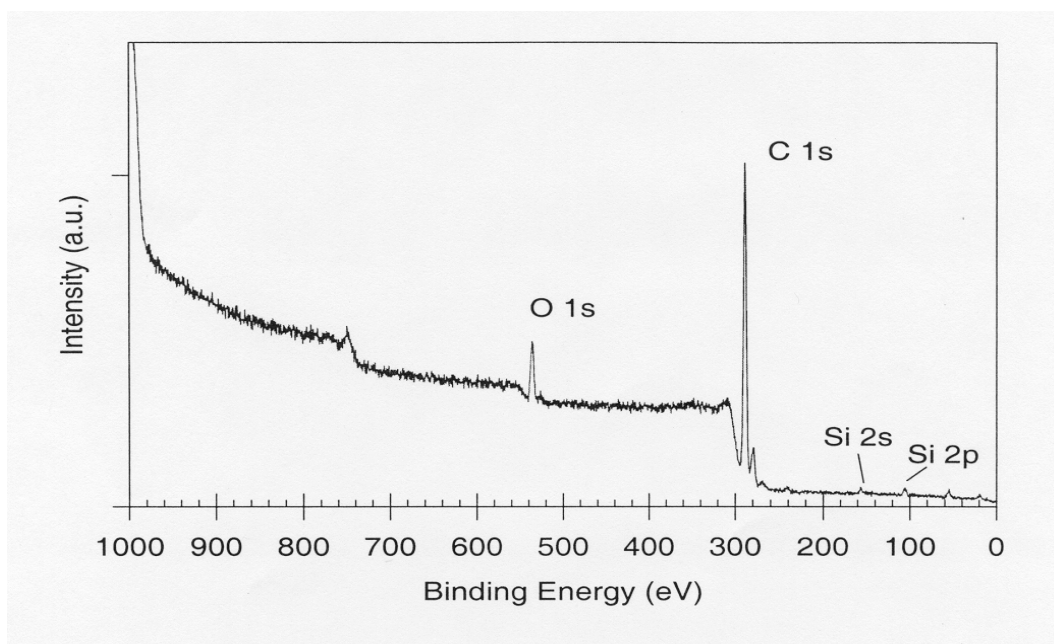
**Figure 11: Lap Joint Bondline of PS/GF (33%) at a Magnification of 1000 Times**



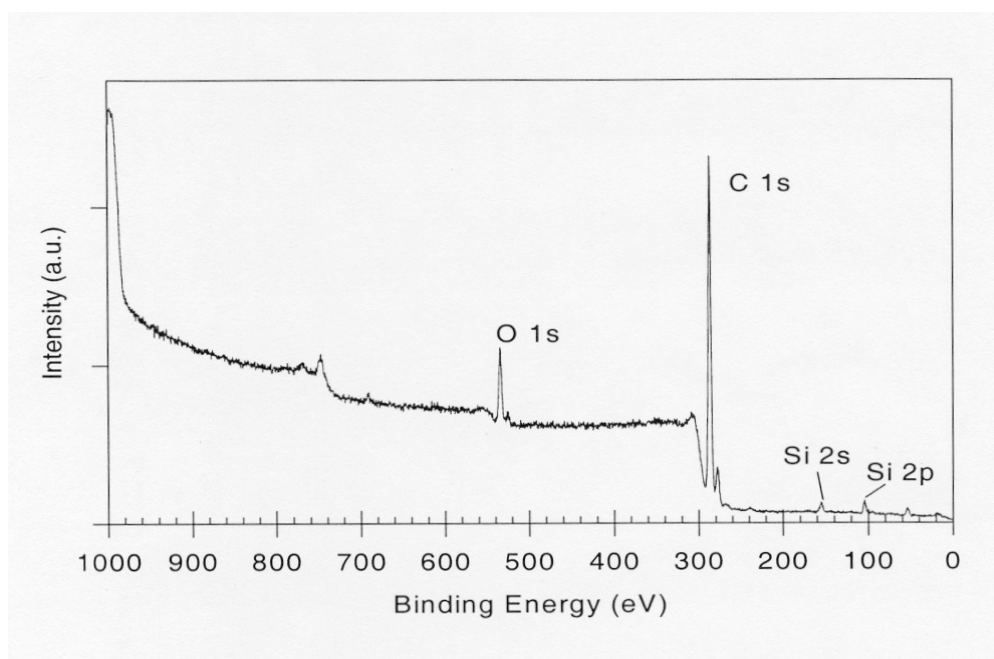
**Figure 12: Locations of Carbon Fibre Filaments on Test Piece**



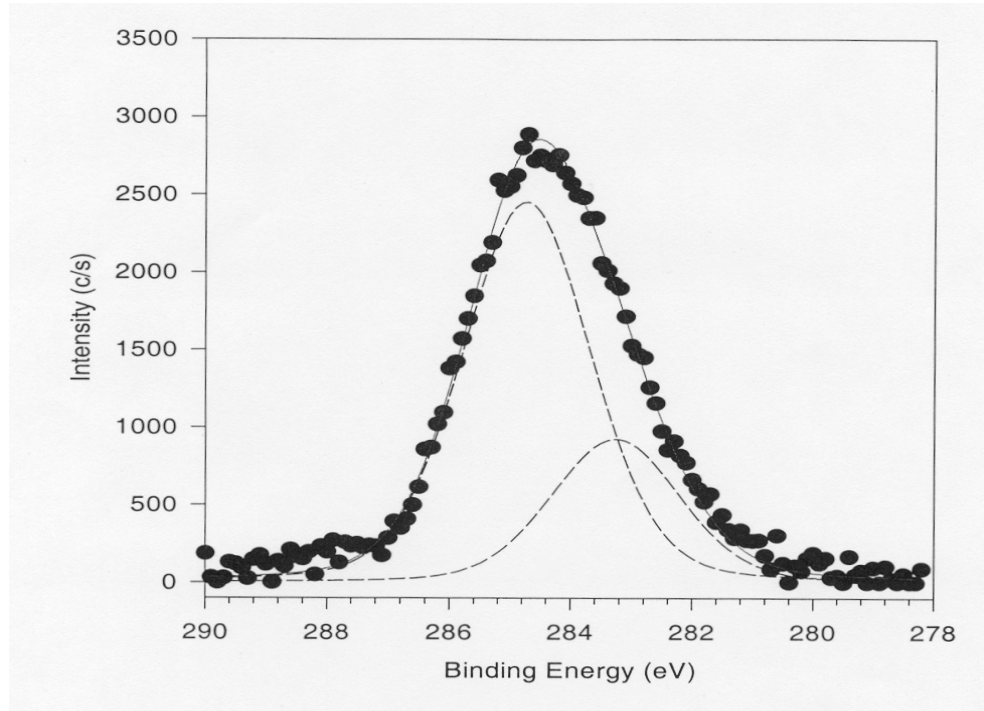
**Figure 13: Locations of Black Marks**



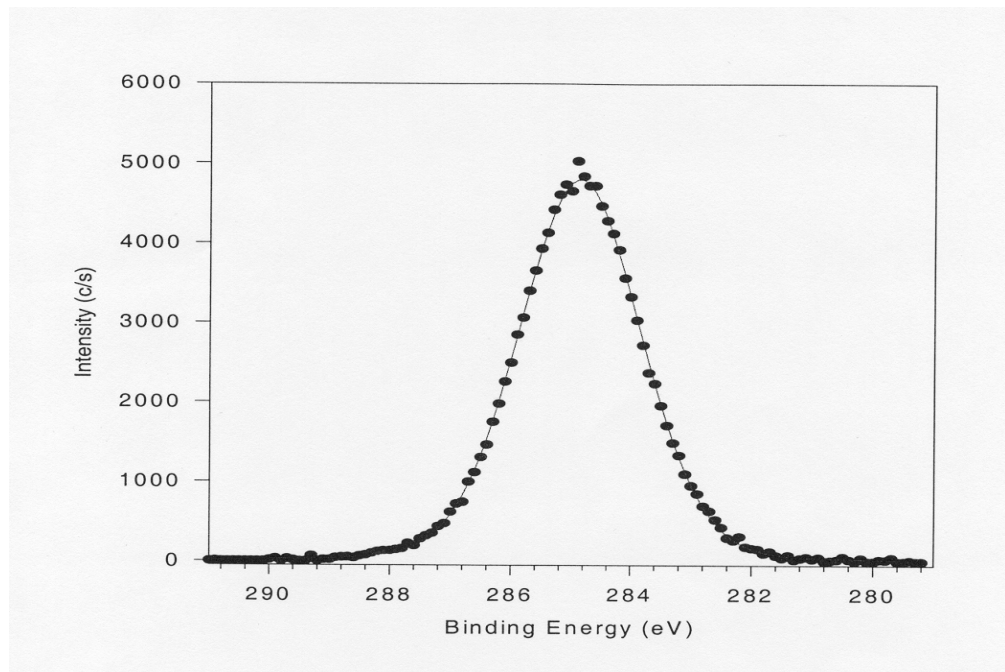
**Figure 14: The Intensity of Elements against Binding Energy in PS/GF (33%) (Microwaved) Sample with Black Patches.**



**Figure 15: The Intensity of Elements against Binding Energy in the Control Sample**



**Figure 16: The Two Peaks of Intensity for Carbon Atoms in the PS/GF (33%) (Microwaved) Sample with Black Patches**



**Figure 17: The Two Peaks of Intensity for Carbon Atoms in the Control Sample with Joining Parameters of VFM, 200 W and 450 seconds**

**Table 1: The Dielectric Constant and Loss Tangent of the Two Matrix Materials of the Composites at 25°C and 3 GHz.**

Matrix Materials	Dielectric Constant	Loss Tangent ( $1 \times 10^{-4}$ )
Low density polyethylene	2.26	3.6
Polystyrene	2.53	5.3